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Pilot demonstration of sugarcane juice ultrafiltration in an Indian sugar factory

A.M. Ghosh, M. Balakrishnan *

Tata Energy Research Institute, Darbari Seth Block, India Habitat Center, Lodhi Road, New Delhi 110003, India

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Abstract

A field demonstration on ultrafiltration of sugarcane juice was conducted in a plantation white sugar factory in northern India. Clarified sugarcane juice at 91–97 °C was processed using polymeric spiral wound membrane modules in a pilot plant with a design capacity of 10 m^3/h. The investigations focused on the feed characteristics, plant performance in terms of the permeate flux and quality and limitations experienced during operation. The trials indicated that the membrane modules were tolerant to elevated temperatures and displayed satisfactory separation with an average purity rise of 0.9 units, 31% lower turbidity and 47% lower color in the permeate. The average flux was, however, low (7 l/m^2 h). The feed quality needs to be controlled, particularly with respect to suspended solids content and additional foulants introduced in the processing scheme. Further, the water available for membrane washing and cleaning was below recommended standards and must be improved.

Keywords: Clarified sugarcane juice; Ultrafiltration; Industrial demonstration; Spiral wound membrane module

1. Introduction

India is the world's largest producer of ''plantation white'' or ''mill white'' sugar by the double sulphitation process (Fig. 1). A major challenge in this manufacturing scheme is to ensure that the clarification step consistently results in a juice of high clarity and low color. However, variations in cane variety, changes in agro-climatic conditions and fluctuations in the manufacturing process leads to non-uniform juice characteristics. Further, the liming–sulphitation process employing conventional clarifier-settlers for removing the precipitated impurities is incapable of eliminating colloidal and dissolved macromolecular substances. For this reason, the clarified juice is typically slightly hazy and has a dark brownish yellow color. Thus the quality of plantation white sugar, characterized by a high color and sulfite content, is noticeably inferior to refined sugar (Schiweck & Clarke, 1994).

Clarification of sugarcane juice by microfiltration and ultrafiltration (UF) has been explored extensively both in laboratory (Bhattacharya, Agarwal, De, & Rama Gopal, 2001; Kishihara, Fujii, & Komoto, 1981, 1983; Kishihara, Tamaki, Fujii, & Komoto, 1989; Nene et al., 2000; Verma, Srikanth, Das, & Venkidachalam, 1996) and factory trials (Cartier, Theoleyre, Lancrenon, & Decloux, 1996; Eringis & Eaton, 2000; Fechter et al., 2001; Kochergin et al., 2000; Kwok, 1996; Madsen, 1973; Nielsen, Kristensen, & Madsen, 1982; Saska, McArdle, & Eringis, 1999; Willet, 1997). It is well established that the permeate obtained by membrane filtration possesses better clarity, lower viscosity and reduced color. Consequent benefits include higher crystal yield (Cartier et al., 1996; Kishihara et al., 1989), energy savings due to reduced evaporator steam consumption (Kwok, 1996) and increased capacity of evaporators, vacuum pans, crystallizers and centrifuges (Eringis & Jaferey, 2001).

In earlier trials conducted in an Indian sugar mill, we investigated the effect of operating parameters, membrane and module properties on the UF of different process streams in plantation white sugar manufacture (Balakrishnan, Dua, & Bhagat, 2000a,b; Balakrishnan, Dua, & Khairnar, 2001; Ghosh, Balakrishnan, Dua, & Bhagat, 2000). This paper presents the experiences of an industrial scale pilot demonstration for the UF of

* Corresponding author. Tel.: +91-11-4682100/4682111x2607; fax: +91-11-4682144/4682145.

E-mail address: malinib@teri.res.in (M. Balakrishnan).

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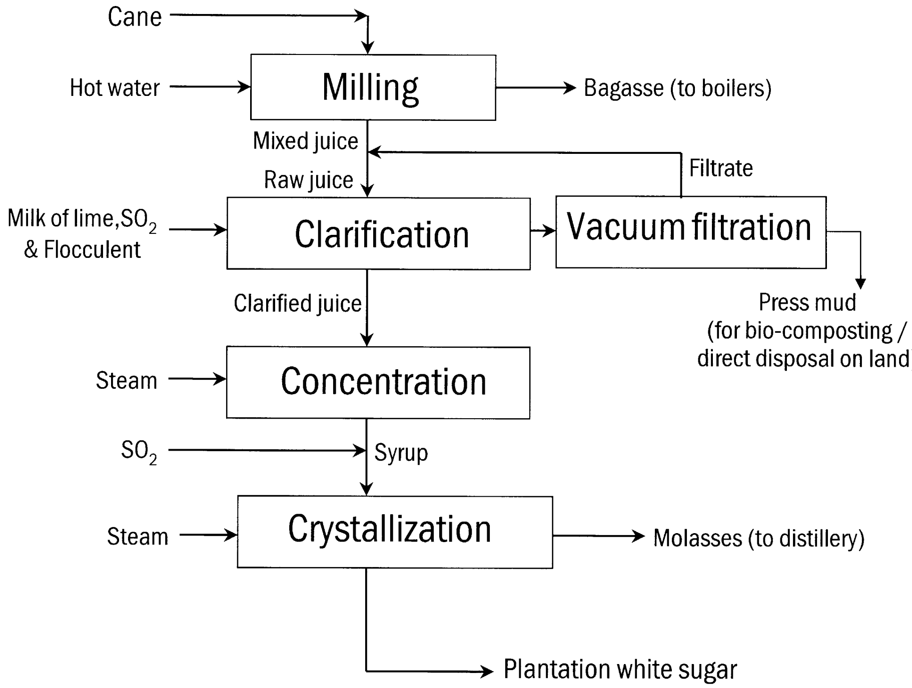


Fig. 1. Schematic of plantation white sugar manufacture by double sulphitation process.

clarified juice. The focus is on the pilot plant performance as well as the limitations encountered during operation.

2. Materials and methods

2.1. Sugarcane juice

The feed to the pilot plant was the hot (91–97 °C) clarified juice overflow from the mills clarifiers. The feed was obtained continuously through a tapping located upstream of the clarified juice heaters, prior to the evaporators.

2.2. Membrane modules

The spiral wound UF membrane modules were procured from Permionics, Vadodara, Gujarat, India. The 8 in. × 40 in. (diameter × length) modules with an effective filtration area of 20.23 m^2/module, employed 20 kD nominal molecular weight cutoff rating polyethersulphone membranes. The modules were tolerant to pH in the 1–14 range and were designed to withstand temperatures up to 110 °C.

2.3. UF demonstration plant

The demonstration was conducted at The Simbhaoli Sugar Mills Limited, Simbhaoli, dist. Ghaziabad, Uttar Pradesh. The pilot installation comprised a pre-filtration set-up followed by the UF assembly, suitably integrated with the mills' process scheme (Fig. 2). The pre-filtration system (Rak Din Engineers, Gurgaon, Haryana, India and in-house fabrication in the mill) for suspended solids removal consisted of 100, 50 and 10 μm stainless steel screens in series followed by 1 μm cartridge. The UF system (Permionics, Vadodara, Gujarat, India) had a total of ten fiber reinforced plastic pressure vessels arranged in parallel with appropriate piping for the feed, permeate, recirculation and bleed streams. Each pressure vessel accommodated four membrane modules in series. The total membrane area of the plant was calculated to be 809 m^2. Separate feed and recirculation pumps (Johnson Pumps, Ahmedabad, Gujarat, India) were provided. The unit was equipped with suitable instrumentation to measure the flow, temperature and pressure and allowed for operation in the feed-and-bleed-mode. All juice/wash streams containing sugar were directed to a common juice sump and the contents were pumped regularly into the mills' raw juice tank. The membrane cleaning chemicals and the associated wash waters were piped to the sewage system for subsequent treatment in the mills' effluent treatment plant.

2.4. Plant operation

The pilot trials consisted of a series of experimental runs comprising the following sequence of steps:

(a) Measurement of pure water permeability of clean membrane.

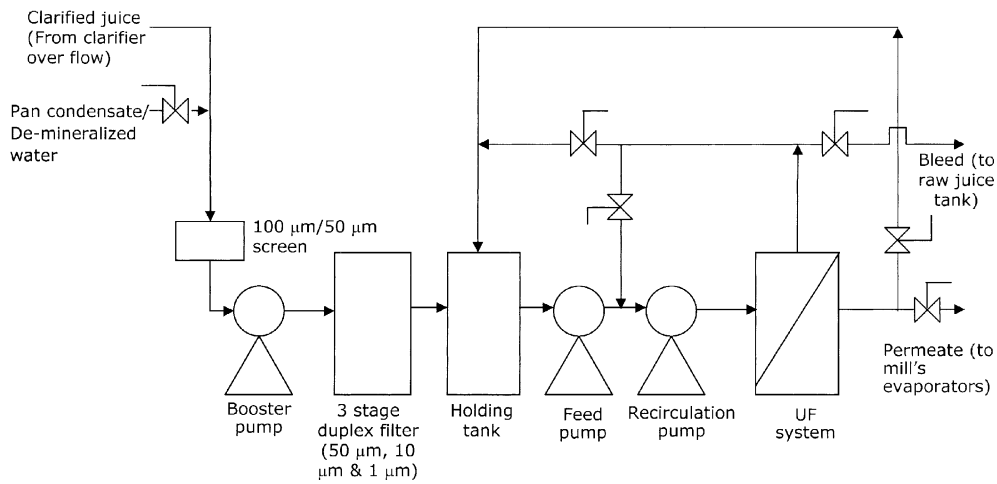


Fig. 2. Schematic of demonstration plant set-up.

(b) Juice UF ("production cycle").
(c) Measurement of pure water permeability of fouled membrane.
(d) Membrane cleaning.

The pure water permeability was measured at about 80 °C using condensate from the vacuum pans ("pan condensate"). The production cycle which typically lasted 8–14 h, commenced when the condensate was replaced with hot clarified juice. The plant was operated in the feed-and-bleed mode during juice UF. The volume concentration factor was between 2 and 6. Operational data such as flow rates, pressures and temperatures were monitored and recorded at regular 15–30 min intervals. Juice samples from the feed, permeate and retentate streams were collected once in 2–3 h for analysis. The plant setting was not disturbed during the course of operation. The production cycle was terminated once the permeate flow rate dropped below 4 m^3/h. The plant was then drained and flushed with pan condensate before measuring the pure water permeability of the fouled membranes. This was followed by chemical cleaning and the membranes were stored overnight in either 0.5–1% formalin or 50–100 ppm hypochlorite. Each experiment lasted 12–18 h. Towards the close of the 2000–2001 crushing season, continuous, round the clock operation was conducted for a limited period of 72 h.

2.5. Analysis

The protocols prescribed by the Sugar Technologists Association of India (Varma, 1988) were followed for juice analysis. The brix and pol were measured using a standardized brix spindle (0–10 or 10–20 brix range, Reige, Germany) and polarimeter (Schmitz and Heinsch, Germany), respectively. The purity rise and the rejection of various juice components was calculated as follows:

$$\text{Purity (\%)} = (\text{Pol per cent juice}/\text{Brix}) \times 100$$

$$\text{Purity rise } (-) = (\text{Purity})_{\text{permeate}} - (\text{Purity})_{\text{feed}}$$

$$\text{Brix rejection (\%)} = \{1 - (\text{Brix})_{\text{permeate}}/(\text{Brix})_{\text{feed}}\} \times 100$$

$$\text{Non-sugars rejection (\%)} = \{1 - \{(\text{Brix} - \text{Pol})_{\text{permeate}}/(\text{Brix} - \text{Pol})_{\text{feed}}\}\} \times 100$$

$$\text{Sugar rejection (\%)} = \{1 - (\text{Pol})_{\text{permeate}}/(\text{Pol})_{\text{feed}}\} \times 100$$

The juice color and turbidity were evaluated spectrophotometrically at 560 and 900 nm, respectively (ICUMSA, 1994). The turbidity was expressed as

$$\text{Turbidity } (S) = 100 \times \text{absorbance at 900 nm}$$

The calcium oxide (CaO) content was estimated by titration against EDTA (Varma, 1988). The sample pH was measured using a calibrated pH meter with automatic temperature compensation (Eutech Cybernetics, Singapore). A conductivity meter (Eutech Instruments, Singapore) was used to estimate the ionic content. Specific inorganics were analyzed by atomic absorption spectrophotometry (AAS) (AAS-5FL, Analytik Jena GmbH, Germany).

The average suspended solids content was estimated by weighing the particulate matter collected over a 50 μm stainless steel screen during the course of the juice UF experiment. The total juice filtered was computed from the feed flow rate and the duration of juice UF. The suspended solids composition was estimated as per the procedures laid down by the Bureau of Indian Standards (1997).

3. Results and discussion

3.1. Feed characteristics

Table 1 summarizes the average feed properties recorded over the course of one crushing season (November 2000 to April 2001). The suspended solids comprised a mixture of fine bagasse ("bagacillo") and precipitated impurities ("mud"). Table 2 presents the composition profile. It was observed that the juice characteristics, particularly the suspended solids content, was dependent upon the following mill operation conditions:

(1) *Clarification parameters:* This included concentration of clarification chemicals (milk of lime, sulfurdioxide and polyelectrolyte flocculent), juice temperature and clarifier retention time. The mill attempted to maintain the sulphited juice temperature at 102 °C; however, variations were observed in the 92–108 °C range. Further, the mill was initially operating the clarifier with a retention time of 70 min as against the recommended time of 150–180 min (2.5–3 h) (Honig, 1963). Consequently, proper settling of the precipitated impurities was not ensured and the particulate content in the clarified juice was correspondingly high (nearly 0.1%).

(2) *Cane crush rate and milling parameters:* Lower crush rates, typically due to temporary cane shortages, resulted in lower bagacillo content. In contrast, suspended solids content increased immediately after mill cleaning and maintenance operations owing to finer cane preparation prior to the milling step.

(3) *Cane variety:* Seasonal and even daily variations in the cane variety were observed. In particular, each year from about mid February to the end of the season, the mill receives a crop of "plant cane". Observations over two crushing seasons between 1999 and 2001 indicate that the plant cane results in a higher juice brix (12.1–16%) as compared to the crop processed in the earlier part of the season (11.9–14.5% brix). Consequently, in the latter half of the season, the mud settling characteristics was relatively poor, often leading to a higher suspended solids content in the clarifier overflow.

Table 1
Feed characteristics (2000–2001 season)

Property	Average value
Suspended solids (%)	0.02
Brix (%)	14.13
Pol (%)	11.46
Purity (%)	80.99
pH (–)	6.99
TDS (ppt)	1.87
Conductivity (mS)	4.07
Turbidity (–)	21.1
Color (absorbance units)	0.91
CaO (mg/l)	1389

Table 2
Profile of suspended solids in clarified juice

Property	Composition (% by mass)
Ash	25
Organic matter	60
Silica (as SiO_2)	7.7
Phosphorus (as P_2O_5)	3.0
Calcium (as CaO)	5.7
Iron (as Fe_2O_3)	0.7
Sulfur (as SO_3)	2.0

3.2. Demonstration plant performance

Fig. 3 describes permeate flux during the course of the demonstration plant operation in the 2000–2001 crushing season. Each data point represents the average flux obtained in one production cycle. The flux profile displayed a characteristic exponential decline approaching a stable value of around 7 l/m^2 h after nearly 60 h of operation. Throughout the trials, the feed temperature was between 91 and 97 °C. The corresponding permeate temperature was lower at 86–90 °C due to heat loss from the system piping which were not lagged. Further, no additional heating was provided in the recirculation line.

Fig. 4 exhibits the average transmembrane pressure (TMP) and the corresponding pressure drop along the module. The TMP at the initiation of the trials was about 1.57 bar. The value increased progressively and stabilized at about 3.04 bar after 60 h of operation. The pressure drop was however almost constant throughout the trials with an average value of 1.01 ± 0.05 bar indicating the absence of clogging in the feed channel spacers by suspended matter. This was subsequently confirmed by a physical examination of the membrane modules at the conclusion of the testing season. The multi-stage pre-filtration therefore appeared to be effective.

Fig. 5(a) and (b) exhibit the brix and purity profiles, respectively, for the feed, permeate and retentate streams. The brix, pol and non-sugars rejection was calculated to be $2.43 \pm 1.41\%$, $1.33 \pm 1.49\%$ and $7.21 \pm 4.70\%$, respectively. The average purity rise with reference to the pre-filtered feed was 0.9 units. A visual examination of the ultrafiltered juice showed that it was sparkling clear. Table 3 summarizes the juice characteristics.

At the conclusion of the season's trials, the membrane modules were removed from the pressure vessels and examined (Leitz, 1996). The modules were physically intact after repeated exposure to hot juice under pressurized conditions. No particulate matter was visible at the entrance of the pressure vessels or on the surface of the module. The modules were stored in 200 ppm

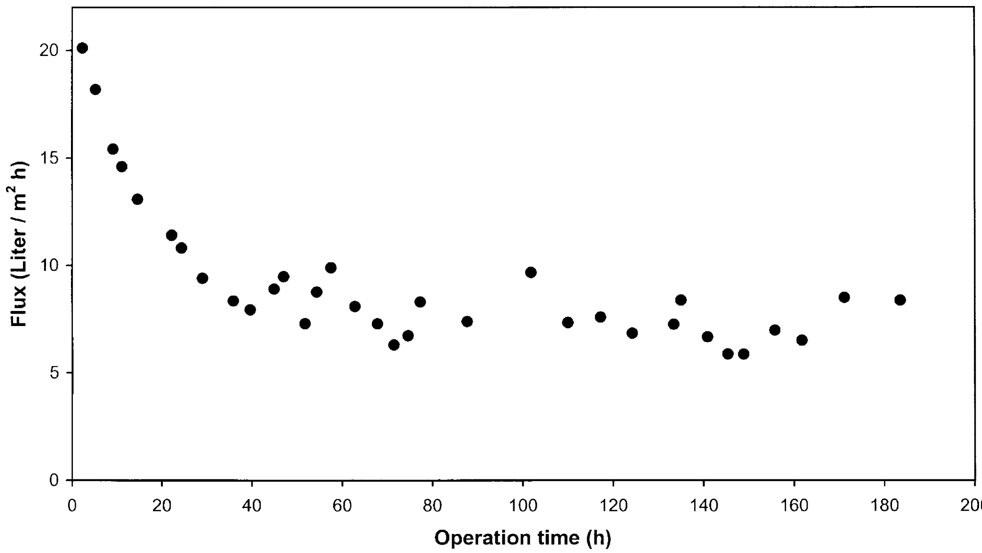


Fig. 3. Performance of demonstration plant: permeate flux.

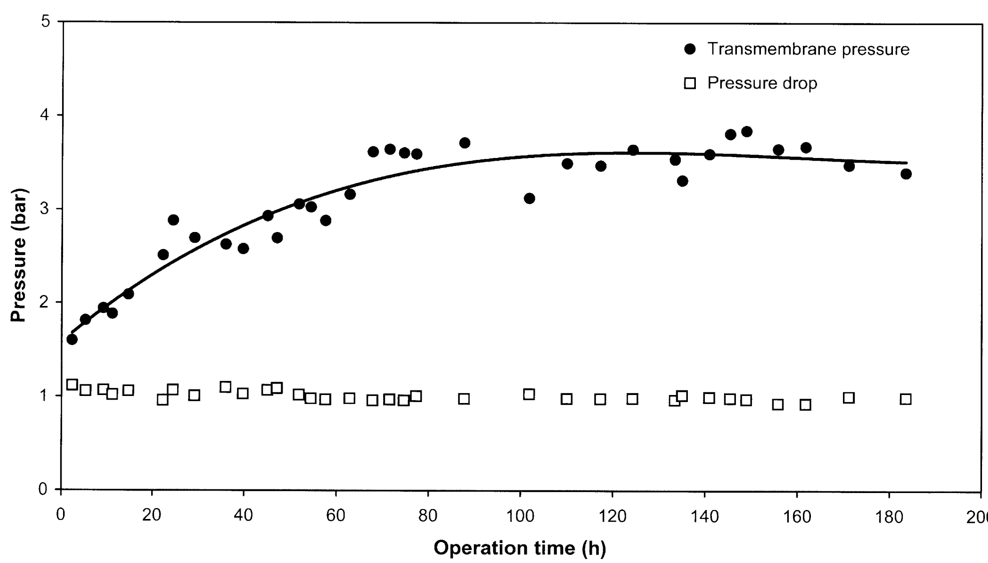
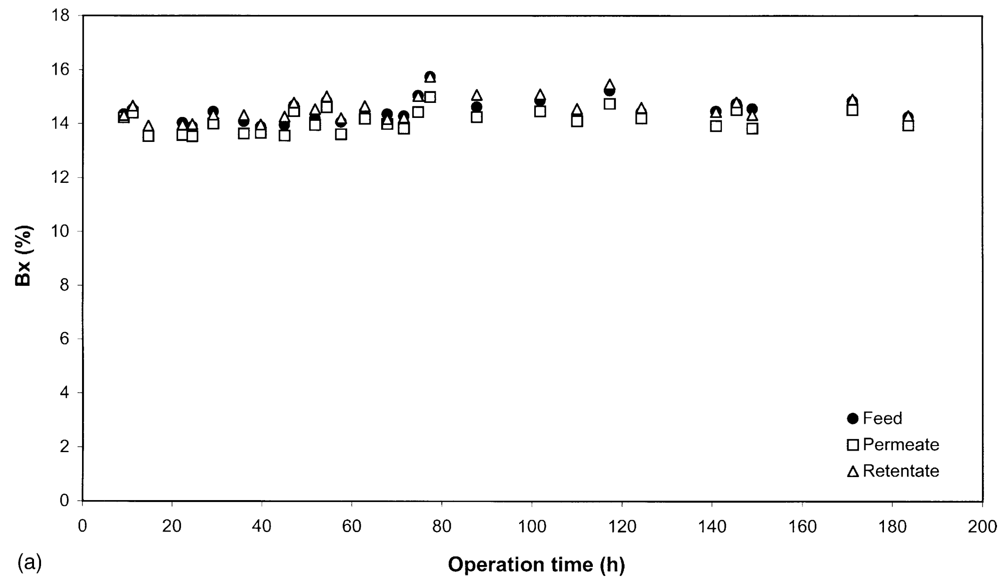


Fig. 4. Variation in TMP and pressure drop during demonstration plant operation.

hypochlorite solution at 18 °C during the off season (May 2001 to November 2001) as per the manufacturers recommendation.

3.3. Critical issues in UF plant operation

(1) *Suspended solids content in juice:* The average suspended solids content in the clarified juice was 0.02% in the 2000–2001 season. However, very high particulate loads (up to 0.2%) were encountered a few times due to disturbances in the clarification operation. The particulate matter comprised both bagacillo and mud. A particle size analysis of the bagacillo indicated that less than 0.2% particles were below 10 μm in diameter (Dhoke, 2000). However, the bagacillo is fibrous, swells upon wetting and agglomerates readily due to the presence of the polyelectrolyte flocculent used to aid the settling of the precipitated impurities. As these particles can clog the module feed channel spacers, an effective pre-filtration system for suspended solids removal becomes crucial. It was observed that a 100 μm stainless steel screen is adequate for removing bagasse particles but a tighter 50 μm screen was required to retain the mud. However, these screens are unlikely to eliminate the colloidal matter that is reported to be in the 0.1–0.5 μm range (Nene et al., 2000). The pre-filtration requirement for limed–sulphited juice appears to be more stringent when compared to that needed for limed



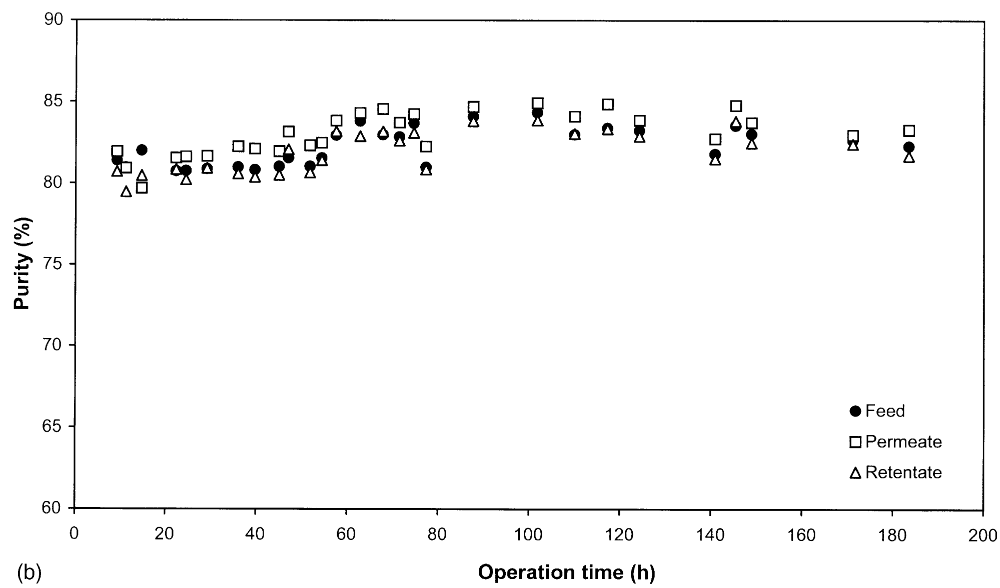


Fig. 5. Performance of demonstration plant: juice characteristics; (a) Brix, (b) Purity.

Table 3
Comparison of average juice properties

Property	Feed	Permeate	Retentate
pH (–)	6.99	6.98	6.98
TDS (ppt)	1.87	1.80	1.79
Conductivity (mS)	4.07	4.05	4.09
Turbidity (–)	21.1	14.5	31.8
Color (absorbance units)	0.91	0.48	1.37
CaO (mg/l)	1389	1344	1411

(defecated) sugarcane juice in raw sugar manufacture where a 100 μm screen is reported to be effective for particulate removal (Saska et al., 1999).

(2) *Feed composition:* The typical composition of sugarcane juice is 75–88% water and 10–21% sucrose, with the balance being reducing sugars, organic matter, nitrogenous substances and inorganic compounds (Mathur, 1986). The non-sugar impurities in the juice such as proteins, polysaccharides, waxes, gums, etc. are known to cause membrane fouling. Apart from these inherent foulants, the presence of additional components introduced during processing is also significant. Bactericide is added at regular intervals at the cane milling station to prevent microbial growth. Cationic polyelectrolyte flocculent is continuously introduced in the clarifiers to accelerate the settling of the precipitated impurities. All holding tanks, piping and pumps are constructed of mild steel; this, in turn, increases the iron content in the juice. Also, traces of grease and lubricant were observed to enter the process stream through

Table 4
Summary of average wash water characteristics

Stream	Temperature (°C)	pH (–)	Conductivity (mS)	TDS (ppt)	Inorganics (mg/l)		
					Fe	Si	Mn
Condensate (sulphited juice heaters)	70–80	9.1	0.10	0.10	NA[a]	NA[a]	NA[a]
Condensate (vacuum pans)	80–90	8.6	0.10	0.10	0.39	1.83	0.015
Raw water	Ambient	8.1	0.55	0.33	0.03	1.30	0.06
Drinking water	Ambient	7.13	1.49	0.95	0.05	1.54	0.44

[a] Not analyzed.

pumps, piping seals, joints, etc. further contributing to the buildup of membrane foulants. In particular, it was observed that the recommended flocculent dosage of 1–3 ppm was often exceeded during operation.

(3) *Production cycle:* One of the major requirements was to maximize the duration of the juice UF between two cleaning cycles. A daily 2 h cleaning cycle was acceptable to the mill. However, the actual juice UF cycles varied between 8 and 14 h depending upon the incoming juice quality. Further, the membrane cleaning protocol also affected the length of the subsequent production cycle. Standard cleaning procedures using acid, alkali and detergent, as recommended by the membrane manufacturer, were tried out. The UF runs lasted longer when alkaline cleaning was employed and the modules were stored overnight in hypochlorite solution.

(4) *Wash water quality:* Condensate from the mills vacuum pans (pan condensate) was used for membrane rinsing and cleaning. It is obtained typically at about 80 °C and is used as feed for the boilers. The average pH, monitored over two crushing seasons from 1999 to 2001, was 8.6 and the conductivity was 0.1 mS. Any shortfall in pan condensate supply was made up with demineralised water at ambient temperature (28–32 °C).

Both the raw water and drinking water available at the mill site had a high dissolved solids content and were therefore considered unsuitable for membrane cleaning. The pan condensate supply was adequate; however, the quality was not always satisfactory with occasionally very high iron and silica content as well as pH fluctuations. The pH variation was linked to problems in controlling the dosage of milk of lime and sulfurdioxide in the juice clarification step. Further, the pan condensate pH was always high (>9.0) following the cleaning of evaporator tubes with hot caustic soda that was carried out once every 45 days.

Table 4 presents an analysis of the various water streams available at the mill. The Indian sugar industry conventionally uses mild steel piping and equipment; thus, it is almost impossible to meet the recommended specification of less than 0.05 mg/l iron (Cheryan, 1998) for the wash water. For instance, an iron content of 0.07 mg/l was observed for a freshly collected pan condensate sample; the value increased dramatically to 2.66 mg/l for a pipe hold-up sample.

(5) *Temperature cycling:* The UF system was subjected to a range of operating temperatures during the trials. The clarified juice processing at 91–97 °C was followed by hot water wash at about 80 °C. The chemical cleaning was performed in the 30–70 °C range. The ambient temperatures during the trials were 28–40 °C in the daytime and 15–28 °C in the night. Except for a short period, the system was not operated during the mills' night shift (12 midnight to 8 a.m.). Thus, the system components experienced cyclic heating and cooling. This resulted in visible contraction in one of the end plates fittings when the system was not in operation; further, polypropylene valves used in the pre-filtration system displayed significant leaks when operating at temperatures below 60 °C during the cleaning cycle.

(6) *Plant automation:* Proper automation is critical for reliable long-term testing and evaluation of the UF pilot plant (Kochergin, 1998). Most Indian sugar mills employ limited automation based usually on an 'island' approach restricted to specific areas of operation. The present trials employed a simple manually operated unit that was adequate to demonstrate the feasibility of membrane filtration on an industrially acceptable scale in the local sugar mill environment. However, for subsequent design of a full-scale plant, automatic control of flow, temperature and pressure is necessary and the pilot plant would have to be accordingly equipped with appropriate controls.

4. Conclusions

Field testing of an UF pilot plant for processing clarified sugarcane juice was successfully completed in a plantation white sugar mill. The spiral wound polymeric membrane modules employed for the trials are satisfactory in terms of high temperature tolerance and superior permeate quality; however, the flux is low and needs to be improved. In particular, the quality of the mills clarified juice, especially the suspended solids content, as well the wash water used for membrane cleaning are critical factors affecting UF performance.

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